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(54) METHOD FOR PRODUCING HIGH STRENGTH  
SINTERED ALLOY STEEL

(57) Abstract:

PROBLEM TO BE SOLVED: To provide a method for producing high strength sintered alloy steel in which the effect of improving mechanical properties by the addition of Ni can sufficiently and stably be exhibited.

SOLUTION: Raw material powder obtained by mixing Fe mixed powder of Fe coarse powder and Fe fine powder with Ni fine powder, Mo fine powder and Mn

powder is compacted to obtain a green compact, next, the compact is sintered to obtain a sintered body, and then, the sintered body is subjected to heat treatment. The composition of the steel contains, by weight, 1.7 to 7.0% Ni, 0.2 to 1.0% Mo, 0.1 to 0.8% Mn, 0.3 to 0.6% C, and the balance Fe with inevitable impurities, and moreover, the average Ni concentration in an Ni-enriched phase in the structure is controlled to 2.5 times or more to the average Ni concentration in the matrix structure.

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最終頁に続く

(54) 【発明の名称】 高強度焼結合金鋼の製造方法

(57) 【要約】

【課題】 N i 添加による機械的特性の向上効果が十分に、かつ安定して発揮される高強度焼結合金鋼の製造方法を提供する。

【解決手段】 F e 粗粉末と F e 細粉末との F e 混合粉末に N i 細粉末、M o 細粉末および M n 粉末を混合してなる原料粉末を圧粉成形して圧粉体を得、次いで該圧粉体を焼結して焼結体を得、次いで該焼結体を熱処理する。鋼の組成を、N i が 1. 0 ~ 7. 0 w t %、M o が 0. 2 ~ 1. 0 w t %、M n が 0. 1 ~ 0. 8 w t %、C が 0. 3 ~ 0. 6 w t %、残部が F e および不可避不純物からなるものとし、さらに、組織中の N i リッチ相の平均 N i 濃度を基体組織の平均 N i 濃度に対して 2. 5 倍以上に調整する。

## 【特許請求の範囲】

【請求項1】 Fe粗粉末とFe細粉末とのFe混合粉末にNi細粉末、Mo細粉末およびMn粉末を混合してなる原料粉末を圧粉成形して圧粉体を得、次いで該圧粉体を焼結して焼結体を得、次いで該焼結体を熱処理することにより、Niが1.0～7.0wt%、Moが0.2～1.0wt%、Mnが0.1～0.8wt%、Cが0.3～0.6wt%、残部がFeおよび不可避不純物からなる組成を有するとともに、組織中のNiリッチ相の平均Ni濃度が基体組織の平均Ni濃度に対して2.5倍以上を示す高強度焼結合金鋼を得ることを特徴とする高強度焼結合金鋼の製造方法。

## 【発明の詳細な説明】

## 【0001】

【発明の属する技術分野】本発明は、高強度焼結合金鋼の製造方法に係り、特に、Fe粉末の混合形態や含有元素の配合を規定することによって、より高レベルな機械的特性（引張り強度や疲労強度等）が要求される高強度焼結合金鋼を好適に製造し得る技術に関する。

## 【0002】

【従来の技術】原料粉末を所望の形状に圧粉成形し、これを焼結、熱処理して得られる高強度焼結合金鋼は、各種産業界で広く使用されており、特に、複雑な形状の機械部品や軸受等の製品に好適である。この高強度焼結合金鋼にあつては、高強度化の要求が益々高まる傾向にあり、それに応える手段として、鋼中にNiを含有させることが知られている。

【0003】鋼中へのNiの含有方法は様々であるが、その1つとして、NiをFe粉中に固溶させたブレアロイFe粉を用いる方法がある。この方法は、圧粉体を焼結した場合にNi濃度が均一になるという点で優れており、また、焼入れ性が良好なため焼入れ後は均一なマルテンサイト組織になり易く、疲労強度が向上するといった利点があった。しかしながら、固溶硬化によって粉末の圧縮性が低下することから機械的特性の低下を招いたり、靱性がやや劣るといった欠点があった。鋼中へNiを含有させる他の方法としては、特開平2-145703号公報によって、Ni、Cu、Moの単体元素、あるいはこれら元素のうち2種以上の元素を予め合金させた合金微粉を、原料粉末に拡散付着させる方法が提案されている。この方法は、上記ブレアロイFe粉を用いる場合に比べれば粉末の圧縮性に優れるものの、Niの合金化による圧縮性の低下は依然として避けられず、しかも拡散付着処理を要することによってコストが上昇するという問題も含んでいる。

【0004】すなわち、上記いずれの方法も、Ni含有粉末を添加させることによって粉末の圧縮性が低下するといった問題を抱えており、これは、鋼の強度を向上させる目的に相反するものである。そこで、この問題を解決することを目的とした技術が、特公平7-45683

号公報によって提案されている。同公報によれば、粒子の大きさが45μm以下のNi、CuおよびMoの合金粉末を、潤滑剤とバインダとの共溶融物によってFe粉末の表面に付着させることにより、圧縮性に優れる粉末を得ることができるとされている。

## 【0005】

【発明が解決しようとする課題】しかしながら、上記解決策では、Niの凝集を効果的に解砕して鋼中にNiを均一に分散させることが困難であり、そのため、機械的特性が大きくばらつくといった問題が生じる。このように、原料粉末中へのNiの添加は鋼の強度向上に有効であることは明白でありながら、その効果を十分に引き出すには至っていないのが現状であった。

【0006】よって本発明の目的は、Ni添加による機械的特性の向上効果が十分に、かつ安定して発揮される高強度焼結合金鋼を好適に製造することができる方法を提供することにある。

## 【0007】

【課題を解決するための手段】本発明者は、原料粉末中への効果的なNi添加方法はもとより、主体となるFe粉末の混合形態に関して鋭意研究を重ねた。その結果、Fe粉末の粒子の大きさを比較的粗大なものと微細なものの2種類とし、さらにNiを含む他の元素の粒子の大きさおよび配合を規定した原料粉末を用いることにより、圧粉体成形時に良好な圧縮性を示し、ひいては機械的特性に優れた高強度焼結合金鋼を得るに至った。よって、本発明はこのような知見に基づいてなされたものであり、Fe粗粉末とFe細粉末とのFe混合粉末にNi細粉末、Mo細粉末およびMn粉末を混合してなる原料粉末を圧粉成形して圧粉体を得、次いで該圧粉体を焼結して焼結体を得、次いで該焼結体を熱処理することにより、Niが1.0～7.0wt%、Moが0.2～1.0wt%、Mnが0.1～0.8wt%、Cが0.3～0.6wt%、残部がFeおよび不可避不純物からなる組成を有するとともに、組織中のNiリッチ相の平均Ni濃度が基体組織の平均Ni濃度に対して2.5倍以上を示す高強度焼結合金鋼を得ることを特徴としている。

【0008】ここで、本発明の粗粉末、細粉末および粉末について定義する。粗粉末は粒子の平均粒径が50～80μm、細粉末は粒子の平均粒径が20μm以下、粉末は粗粉末と細粉末の間であつて、粒子の平均粒径が20μmを超え、かつ50μmを下回るものとする。

【0009】本発明によれば、Fe粉末を粗粉末と細粉末との組み合わせで構成することにより、圧粉体成形時の圧縮性が良好となり、製品の高密度化が達成される。これは、隣接するFe粗粉末の粒子の間にFe細粉末の粒子が緻密に埋まり、結果として空孔率が大幅に低減することによる。また、原料粉末中に上記添加量でNi細粉末が添加されることにより、製造後の鋼にあつては硬度の高いNiリッチ相が分散する不均質組織が得られ、

この組織が疲労特性、特に亀裂伝播の挙動に大きい影響を及ぼす。すなわち、Niリッチ相は非常に硬いマルテンサイト組織であり、このNiリッチ相により亀裂の伝播が阻害され、迂回による亀裂の偏向や亀裂の停留を引き起こす。このため、引張り強度および疲労強度に優れた高強度焼結合金鋼を得ることができる。さらに、Ni細粉末の添加量を上記範囲内で調整することにより、硬さや強度を制御することができる。本発明では、特に原料粉末を射出成形することなく、単なる圧粉成形によって圧粉体を得るので、コストの上昇が抑えられるといった利点も有する。

【0010】次に、上記各元素の添加量およびNiリッチ相の平均Ni濃度に関する数値限定の根拠を説明する。

Ni: 1.0~7.0wt%、Niは、靱性および強度を向上させる元素として重要であり、含有量が1.0wt%未満では靱性の向上が望めない。一方、7.0wt%を超えると強度の向上効果は飽和する。よって、Niの含有量は1.0~7.0wt%とした。

【0011】Mo: 0.2~1.0wt%

Moは、固溶強化および焼入れ性を高め、また、結晶粒を微細化して機械的特性の向上に寄与する元素であり、含有量が0.2wt%未満ではこれらの効果が望めない。一方、1.0wt%を超えると強度の向上効果は飽和する。よって、Moの含有量は0.2~1.0wt%とした。

【0012】Mn: 0.1~0.8wt%

Mnは、焼入れ性を高めて強度を向上させる作用を有する。Mnが0.1wt%未満では焼入れ性の向上が望めず、0.8wt%を超えると靱性が低下する。よって、Mnは0.1~0.8wt%とした。

【0013】C: 0.3~0.6wt%

Cは強度を向上させる代表的な元素であり、含有量が

$$Ms(^{\circ}C) = (823 - 361(C \text{ wt}\%) - 39(Mn \text{ wt}\%) - 5(Mo \text{ wt}\%) - 17(Ni \text{ リッチ相の} Ni \text{ wt}\%)) - 273 \quad \cdots (1)$$

【0017】Ms点は、熱処理（焼戻し）プロセスの冷却時にマルテンサイトを生成させる重要な温度ポイントである。Ms点は、低温になるにしたがってマルテンサイトの生成量が減少するため、なるべく高温側に設定されることが望ましいが、300℃を超えるとNiリッチ相のNi量が少なくなってNiリッチ相が減少し、機械的特性の向上が望めない。一方、Ms点が0℃未満ではマルテンサイトの生成量が少なくなって残留オーステナ

$$\text{硬さ勾配}(\Delta Hv) = (Ni \text{ リッチ相の} Hv \text{ 硬さ}) - (\text{基地組織の} Hv \text{ 硬さ}) \quad \cdots (2)$$

このようにNiリッチ相と基地組織の硬さ勾配を調整することにより、前述した不均質組織が確保される。硬さ勾配は、Niリッチ相におけるNi濃度、ひいてはNiの添加量によって調整することができる。

【0019】

【実施例】以下、具体的な実施例により本発明をさらに

0.3wt%未満では強度向上の効果が望めない。Cが0.6wt%を超えると過剰なCが析出して強度向上を阻害する。よって、Cは0.3~0.6wt%とした。

【0014】Niリッチ相の平均Ni濃度が基地組織の平均Ni濃度の2.5倍以上

本発明においては、Niリッチ相のNi濃度が基地組織のNi濃度よりも高いことが不均質組織を生成させる上で必須であり、2.5倍以上の数値が確保されていれば、Niリッチ相と基地組織の硬さに明確な差異、すなわち硬さ勾配を現出させることができる。本発明者は、Ni細粉末を2%添加した場合の本発明の鋼につき、熱処理温度を変えて硬さ試験を行ったところ、Niリッチ相のNi濃度が5~15%で、Niリッチ相と基地組織に明確な硬さ勾配が生じることが認められ、その際の基地組織のNi濃度はNiリッチ相のNi濃度の概ね1/2.5であった。よって、Niリッチ相の平均Ni濃度が基地組織の平均Ni濃度の2.5倍以上であることを本発明の特徴とする。

【0015】さて、本発明では、前述したFe混合粉末におけるFe粗粉末とFe細粉末との重量比を、Fe粗粉末:Fe細粉末=5~9:5~1とすることを好ましい形態としている。Fe粗粉末が多すぎると空孔率が増加して密度が低くなり、逆にFe細粉末が多すぎると圧粉成形時の流動性すなわち圧縮性が低下する。Fe粗粉末:Fe細粉末=5~9:5~1とすることにより、隣接するFe粗粉末の粒子の間にFe細粉末の粒子が緻密に埋まり、結果として空孔率が大幅に低減して高密度化が図られ、しかも、良好な圧縮性が確保される。

【0016】また、本発明では、組織中のNiリッチ相の平均Ni濃度を5~15%に調整するとともに、下記(1)式で求められるMs点（マルテンサイト変態開始点）を100~300℃に調整することを好ましい形態としている。

イト量が増加するため、機械的特性の向上が望めない。Niリッチ相のNi濃度はMs点と深く関わっており、Ni濃度が5%未満ではMs点が300℃を超え、Ni濃度が15%を超えるとMs点が100℃を下回る。

【0018】さらに本発明では、Niリッチ相の平均硬さが基地組織よりも硬く、下記(2)式で求められる硬さ勾配を、Hv硬さで30~200に調整することを好ましい形態としている。

詳細に説明する。

【実施例1】Fe粗粉末+Fe細粉末、Ni 2%

300~200メッシュで分級したアトマイズFe粉末（粒径:53~74μm）と、カーボニルFe粉末（平均粒径:5μm）とを、重量比7:3で混合しFe粉末を得た。このFe粉末に、カーボニルNi細粉末（平均

粒径：5 $\mu$ m)、Mo細粉末（平均粒径：5 $\mu$ m以下）、FeMn粉末（平均粒径：20～40 $\mu$ m）およびC（黒鉛）粉末を、Ni：2wt%、Mo：0.5wt%、Mn：0.2wt%、C：0.4wt%の組成になるよう混合させた。さらに、この混合粉末に潤滑剤としてステアリン酸亜鉛粉末を0.75wt%混合させ、実施例1の原料粉末を得た。次いで、この原料粉末に適宜量のメチルアルコール溶液を潤滑剤として添加してから、原料粉末を金型に充填し、686MPaの圧力で加圧して圧粉体を成形した。次いで、圧粉体を水素と窒素の混合ガス雰囲気調整された加熱炉内において1250℃で60分間加熱して焼結し、炉冷した。次いで、900℃のアルゴンガス雰囲気中で30分間の溶体化処理を施した後、油中に投入して焼入れし、同雰囲気中で200℃、60分間の焼戻しを施した。これにより、実施

例1の鋼を得た。

#### 【実施例2】Fe粗粉末+Fe細粉末、Ni6%

Niの含有量を6wt%にした以外は実施例1と同様にして、実施例2の鋼を得た。

#### 【0020】【比較例1】Fe粗粉末、Ni2%

Fe粉末として、-200メッシュで分級したアトマイズFe粉末（粒径：74 $\mu$ m以下）のみを用いた以外は実施例1と同様にして、比較例1の鋼を得た。

#### 【比較例2】熱処理せず鍛造

焼結後、鍛造処理を行った以外は実施例1と同様の熱処理を施して、比較例2の鋼を得た。上記各実施例および各比較例のFe粉末およびニッケル粉末の仕様を表1に、また、各元素の組成を表2にそれぞれ示す。

#### 【0021】

【表1】

	成 分 (wt%)				
	Ni	Mo	Mn	C	Fe
実施例1, 比較例1, 2	2.0	0.5	0.2	0.4	Bal
実施例2	6.0	0.5	0.2	0.4	Bal

20

#### 【0022】

【表2】

	Fe粉末		Ni
	粗粉末	細粉末	細粉末
実施例1, 2	アトマイズFe粉末	カーボンFe粉末	カーボンNi粉末
比較例2	粒径53～74 $\mu$ m	平均粒径5 $\mu$ m	平均粒径5 $\mu$ m
比較例1	アトマイズFe粉末		カーボンNi粉末
	平均粒径74 $\mu$ m		平均粒径5 $\mu$ m

#### 【0023】A. 機械的特性、硬さ等

上記実施例および各比較例の鋼につき、引張試験および疲労試験を行って機械的特性を調べた。また、相対密度を調べるとともに、Niリッチ相と基地組織の硬さを調

べ、硬さ勾配を算出した。これらの結果を、表3に示す。

#### 【0024】

【表3】

	引張り 強度 (MPa)	疲労強度 (MPa)	耐久限度 比 (R)	伸び (%)	相対密度 (%)	硬さ (Hv)		硬さ勾配 ( $\Delta$ Hv)
						R	M	
実1	1400	410	0.29	2	94	665	615	50
実2	1770	438	0.25	9	96	672	638	34
比1	1300	360	0.28	2	91	662	575	87
比2	816	446	0.55	9	99	241		0

※硬さの「R」はNiリッチ相の硬さ、「M」は基地組織の硬さ

【0025】表3によれば、本発明に基づく各実施例の鋼の硬さ勾配は、比較例と比べると大きく、また、機械的特性では、特に引張り強度に優れていることが判る。硬さ勾配が大ききことにより、不均質組織が生成されて亀裂の伝播を阻害する作用が十分に発揮され、機械的特性が向上したことが推察された。なお、比較例2では焼結後に鍛造を行ったため、組織が混練されてNiリッチ相が生じていない。このため、硬さと引張り強度が他と比べてかなり低下した。

#### 【0026】B. 金属組織観察

次に、上記実施例1の鋼と比較例1の鋼につき、内部組

織を顕微鏡で調べた。図1、図2は、それぞれ実施例1、比較例1の各鋼の内部組織を示す顕微鏡写真である。両者においては、Aで示す白い部分のNiリッチ相を残存したまま、Bで示す基地組織が焼戻しマルテンサイト組織となっていることが認められる。しかしながら、実施例1は比較例1に比べNiリッチ相が小さく、かつ多数分散しており、亀裂の伝播を阻害しやすく、もって鋼の強度が高まることが理解される。

【0027】図3、図4は、それぞれ実施例1、比較例1の各鋼の疲労亀裂伝播経路を示す顕微鏡写真であり、写真の左側の黒い部分が亀裂である。これらの図から不

均質組織による疲労亀裂伝播の違いが明らかである。すなわち、図3では、疲労亀裂の進路はNiリッチ相を迂回するように伝播しており、亀裂の伝播に大きなエネルギーが消費されたことを示している。一方、図4では、Niリッチ相は観察されるものの、その量、大きさともに小さく、亀裂伝播を抑制する効果が少ない。よって、図4では、亀裂が基体組織を伝播し易く、破断し易くなっている。このことから、図3に示す組織の方が図4に比べて強度が高いことがわかる。

【0028】

【発明の効果】以上説明したように本発明においては、高強度焼結合金鋼を製造するにあたって、原料粉末の主体となるFe粉末を粗粉末と細粉末との組み合わせで構

成し、さらに、Niおよび他の元素の組成ならびに粉末の大きさを適切に規定したことにより、引張り強度や疲労強度等の機械的特性に優れた高強度焼結合金鋼を好適に製造することができるという効果を奏する。

【図面の簡単な説明】

【図1】 本発明の実施例の内部組織を示す顕微鏡写真である。

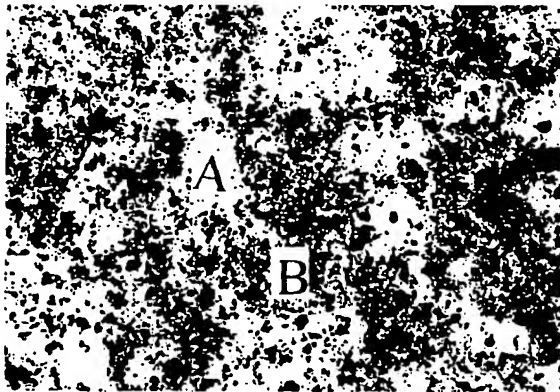
【図2】 比較例の内部組織を示す顕微鏡写真である。

【図3】 本発明の実施例の疲労亀裂伝播経路を示す顕微鏡写真である。

【図4】 比較例の疲労亀裂伝播経路を示す顕微鏡写真である。

【図1】

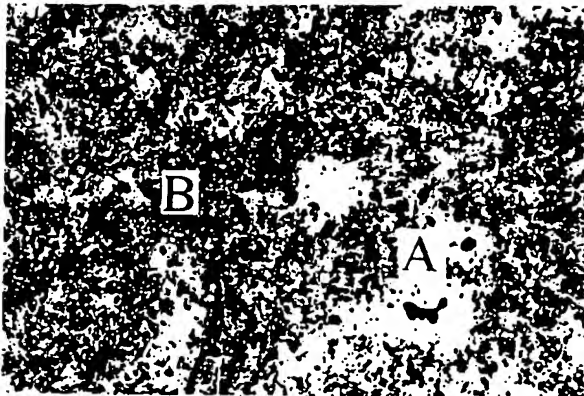
図面代用写真



200 μm

【図2】

図面代用写真



200 μm

【図3】

図面代用写真



【図4】

図面代用写真



フロントページの続き

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To be continued to the last page.

(54) [Title of the Invention]

A manufacturing method of high strength sintered alloy steel

(57) [Abstract]

[Problem to be Solved] To provide a manufacturing method of high strength sintered alloy steel with sufficiently and stably enhanced mechanical properties due to the addition of Ni.

[Means for Solving the Problem] Raw material powder produced as a result of mixing Fe mixed powder made up of Fe coarse powder and Fe fine powder with Ni fine powder, Mo fine powder, and Mn powder is compacted to obtain a compact, the compact is next sintered to obtain a sintered compact, and the sintered compact is then subjected to heat treatment. The steel composition consists of 1.0-7.0 wt% Ni, 0.2-1.0 wt% Mo, 0.1-0.8 wt% Mn, and 0.3-0.6 wt% C, with the balance made up of Fe and inevitable impurities; moreover, the average Ni concentration in the Ni enriched phase of the structure is controlled to be 2.5 times or greater than the average Ni concentration in the matrix structure.

**[Claims]**

**[Claim 1]** A manufacturing method of high strength sintered alloy steel, wherein high strength sintered alloy steel with a composition consisting of 1.0-7.0 wt% Ni, 0.2-1.0 wt% Mo, 0.1-0.8 wt% Mn, and 0.3-0.6 wt% C, with the balance made up of Fe and inevitable impurities, and at the same time having an average Ni concentration in the Ni enriched phase of the structure of 2.5 times or greater than the average Ni concentration in the matrix structure, is obtained via the production of a compact by compacting raw material powder produced as a result of mixing Fe mixed powder made up of Fe coarse powder and Fe fine powder with Ni fine powder, Mo fine powder, and Mn powder; subsequently obtaining a sintered compact by sintering the compact, and then performing heat treatment on the sintered compact.

[Detailed Description of the Invention]

[0001]

[Technical Field of the Invention] The present invention comprises a manufacturing method of high strength sintered alloy steel, and particularly relates to a method that makes it possible to suitably manufacture high strength sintered alloy steel for which higher level mechanical properties (tensile strength, fatigue strength, etc.) are required by regulating a mixed formation of Fe powder and a combination of the contained elements.

[0002]

[Prior Art] High strength sintered alloy steel obtained by compacting raw material powder into a desired shape that is to be sintered and subjected to heat treatment is used widely in various industries, and is particularly suited for products such as mechanical parts with complicated shapes, bearings, etc. With regard to the present high strength sintered alloy steel, demand for high strength is a tendency that is increasing more and more, in response to which, the incorporation of Ni into steel is a means that is well known.

[0003]

The methods of incorporating Ni into steel vary and one particular method uses pre-alloyed Fe powder in which solved Ni remains solid in the Fe powder. This method is excellent in that the Ni concentration becomes uniform when a compact is sintered, and furthermore, because the hardenability is favorable, a uniform martensite structure is easily formed after hardening, resulting in an advantageous enhancement in fatigue strength. However, since the compressibility of the powder deteriorates due to hardening of the solid solution, there have been drawbacks such as deterioration in the mechanical properties or slight inferiority in its toughness. As another method of incorporating Ni into steel, in Japanese Unexamined Patent Application Publication No.2-145703, a method of diffusing and sticking the elements Ni, Cu, Mo, or a pre-alloyed alloy fine powder of more than two kinds from among these elements onto the raw material powder is proposed. This method is excellent in terms of compressibility of the powder compared to the case in which said pre-alloyed Fe powder is used; however, deterioration of the compressibility due to alloy processing of Ni remains unavoidable; moreover, a problem of increased costs which accompany the required treatments of diffusing and sticking remains therein.

[0004]

That is, both of said methods are problematic in that they involve the deterioration of the compressibility of the powder caused by adding powder containing Ni, a

characteristic which is contradictory to the purpose of enhancing steel strength. For this, a method with the aim of solving this problem has been proposed in Japanese Examined Patent Publication No. 7-45683. According to the publication, by sticking alloy powder containing Ni, Cu, and Mo with a grain size of 45  $\mu\text{m}$  or smaller onto the surface of Fe powder via a co-melted lubricant and binder, powder that is excellent in compressibility is apparently obtainable.

[0005]

[Problem the Invention is Intended to Solve] However, with said solution it is difficult to diffuse Ni in the steel uniformly through the effective crushing of Ni flocculation, thus resulting in a problem of large variations in mechanical properties. As described, although adding Ni into the raw material powder is theoretically effective in the enhancement of steel strength, the effect has not been fully realized in reality.

[0006]

Therefore, the purpose of the present invention is to provide a method for suitably manufacturing high strength sintered alloy steel with sufficiently and stably enhanced mechanical properties due to the addition of Ni.

[0007]

[Means for Solving the Problem]

The inventors further conducted intense research on the mixed formation of the main Fe powder as well as an effective method of adding Ni into the raw material powder. As a result, with two kinds of Fe powder distinguished in terms of their grain size, relatively coarse large ones and fine small ones, and further, by using raw material powder for which the grain size and the combination of other elements including Ni were regulated, high strength sintered alloy steel that showed favorable compressibility in forming a compact, and moreover, that was excellent in mechanical properties, was finally obtained. Therefore, the present invention is based on such knowledge and is characterized in that high strength sintered alloy steel having a composition consisting of 1.0-7.0 wt% Ni, 0.2-1.0 wt% Mo, 0.1-0.8 wt% Mn, and 0.3-0.6 wt% C, with the balance made up of Fe and inevitable impurities, and at the same time showing an average Ni concentration in the Ni enriched phase of the structure of 2.5 times or more than the average Ni concentration in the matrix structure, is obtained via the production of a compact by compacting raw material powder produced as a result of mixing Fe mixed powder of Fe coarse powder and Fe fine powder with Ni fine powder, Mo fine powder, and Mn powder, subsequently obtaining a sintered compact by sintering the compact, and then performing heat treatment on the sintered compact.

[0008]

Herein, the coarse powder, fine powder, and powder in the present invention are defined. Coarse powder has an average grain size between 50-80  $\mu\text{m}$ , fine powder has an average grain size that is 20  $\mu\text{m}$  or smaller, and powder is between the coarse powder and the fine powder with an average grain size that is larger than 20  $\mu\text{m}$  but smaller than 50  $\mu\text{m}$ .

[0009]

According to the present invention, composing Fe powder with a combination of coarse powder and fine powder realizes favorable compressibility when forming a compact, and thus, a high density product is achievable. This is due to the large decrease in the porosity rate resulting from the grains of Fe fine powder densely filling in the spaces between the grains of adjacent Fe coarse powder. Moreover, by adding Ni fine powder of said quantity into the raw material powder, as for the produced steel, an inhomogeneous structure is obtainable wherein Ni enriched phases with high hardness are diffused, and the structure has a large influence on fatigue properties, particularly on crack propagation tendencies. In other words, the Ni enriched phase is an extremely hard structure, and with this Ni enriched phase, crack propagation is inhibited, thus causing crack deflections due to the diversion and nonpropagation of cracks. As a result, high strength sintered alloy steel that is excellent in tensile strength and fatigue strength may be obtained. Furthermore, the hardness or strength may be adjusted by controlling the quantity of Ni fine powder added within said range. The present invention also has an advantage in that costs can be kept down due to compacts being obtained by simply compacting the raw material powder, as opposed to using special injection-molding.

[0010] Next, a rationale for the figure limitation with regard to the quantity of said each element to be added and the average Ni concentration of the Ni enriched phase is given.

Ni: 1.0-7.0 wt%

Ni is important as an element to enhance toughness and strength; if the content is less than 1.0 wt%, enhancement of toughness cannot be expected. On the other hand, if exceeding 7.0 wt%, the strength enhancement effect is saturated. Therefore, the Ni content was set to be 1.0-7.0 wt%.

[0011] Mo: 0.2-1.0 wt%

Mo is an element that enhances the strengthening of a solid solution and hardenability, and contributes to the enhancement of mechanical properties by finely processing crystal grains; if the content is less than 0.2 wt% these effects cannot be expected. On the other hand, exceeding 1.0 wt% will saturate the strength enhancement effect. Therefore, the Mo content was set to be 0.2-1.0 wt%.

[0012] Mn: 0.1-0.8 wt%

Mn acts to enhance strength by increasing hardenability. If Mn is less than 0.1 wt%,

enhancement of hardenability cannot be expected; however, exceeding 0.8 wt% will cause a deterioration in toughness. Therefore, Mn was set to be 0.1-0.8 wt%.

[0013] C: 0.3-0.6 wt%

C is a representative element to enhance strength; if the content is less than 0.3 wt%, the strength enhancement effect cannot be expected. On the other hand, if C exceeds 0.6 wt%, the excess C precipitates and inhibits strength enhancement. Therefore, C was set to be 0.3-0.6 wt%.

[0014] The average Ni concentration in the Ni enriched phase is 2.5 times or more than the average Ni concentration in the matrix structure.

In the present invention, it is essential that the Ni concentration in the Ni enriched phase be higher than the Ni concentration in the matrix structure for generating an inhomogeneous structure; if a figure of 2.5 times or more is maintained, a clear difference in hardness between the Ni enriched phase and the matrix structure which serves as a hardness gradient may be realized. The inventors conducted a hardness test with various heat treatment temperatures on the steel of the present invention into which Ni fine powder is added at 2%, and when the Ni concentration of the Ni enriched phase was 5-15 %, it was acknowledged that a clear hardness gradient between the Ni enriched phase and the matrix structure was generated, and the Ni concentration of the matrix structure thereof was approximately 1/2.5 of the Ni concentration in the Ni enriched phase. Hence, the present invention is characterized in that the average Ni concentration in the Ni enriched phase is 2.5 times or more than the average Ni concentration in the matrix structure.

[0015] Further, in the present invention, designating the weight ratio between Fe coarse powder and Fe fine powder in said Fe mixed powder as Fe coarse powder : Fe fine powder = 5-9 : 5-1 is regarded as a preferred formation. An excessive amount of Fe coarse powder will increase the porosity rate, thus lowering the density; on the other hand, an excessive amount of Fe fine powder will lower the fluidity or compressibility when compacting powder. By designating the ratio as coarse powder : Fe fine powder = 5-9 : 5-1, the grains of Fe fine powder densely fill in the spaces between the grains of adjacent Fe coarse powder and, as a result, the porosity rate is largely reduced, thus achieving high density while at the same time maintaining favorable compressibility.

[0016] Moreover, in the present invention, controlling the Ms point (martensite transformation start point) obtained by the following formula (1) at 100-300 °C as well as controlling the average Ni concentration in the Ni enriched phase in the structure to be 5-15 % is regarded as a preferred formation.

$$M_s(^{\circ}C)=(823-361(C\text{ wt\%})-39(Mn\text{ wt\%})-5(Mo\text{ wt\%})-17(Ni\text{ wt\% of a Ni enriched phase}))-273 \text{ ---(1)}$$

[0017] The  $M_s$  point is an important temperature point to generate martensite during cooling in the heat treatment (tempering) process. Regarding the  $M_s$  point, as the temperature goes down, the generated quantity of martensite decreases, thus making it ideal to set the temperature as high as possible; however, if it goes beyond 300 °C, the Ni quantity in the Ni enriched phase decreases and enhancement of the mechanical properties cannot be expected. On the other hand, if the  $M_s$  point is less than 0 °C, the generated quantity of martensite decreases, the quantity of remaining austenite increases, and enhancement of the mechanical properties cannot be expected. The Ni concentration in the Ni enriched phase is closely related to the  $M_s$  point in that if the Ni concentration is less than 5 %, the  $M_s$  point goes beyond 300 °C, but if the Ni concentration goes beyond 15 %, the  $M_s$  point falls below 100 °C.

[0018] Furthermore, in the present invention, controlling the average hardness of the Ni enriched phase to be harder than the matrix structure and the hardness gradient obtained by the following formula (2) to be 30-200 in terms of Hv hardness, is regarded as a preferred formation.

$$\text{Hardness gradient } (\Delta Hv) = (\text{Hv hardness of a Ni enriched phase}) - (\text{Hv hardness of the matrix structure}) \text{ ---(2)}$$

As described, by controlling the hardness gradient of the Ni enriched phase and matrix structure, said inhomogeneous structure may be maintained. The hardness gradient may be controlled by the Ni concentration in the Ni enriched phase and, moreover, by the quantity of Ni added.

[0019]

[Embodiment] Hereinafter, the present invention is further explained in detail based on concrete embodiments.

[Embodiment 1] Fe coarse powder + Fe fine powder, Ni 2%

Atomized Fe powder (grain size: 53-74  $\mu\text{m}$ ) classified by 300-200 mesh and Carbonyl Fe powder (average grain size: 5  $\mu\text{m}$ ) were mixed at a weight ratio of 7:3 to obtain Fe powder. Into the Fe powder, Carbonyl Ni fine powder (average grain size: 5  $\mu\text{m}$ ), Mo fine powder (average grain size: 5 $\mu\text{m}$  or smaller), Fe Mn powder (average grain size: 20-40  $\mu\text{m}$ ) and C (granite) powder were mixed so as to obtain the composition Ni: 2 wt%, Mo: 0.5 wt%, Mn: 0.2 wt%, and C: 0.4 wt%. Furthermore, into the mixed powder, 0.75 wt% of zinc stearate powder was mixed as a lubricant to obtain the raw material



powder of Embodiment 1. Next, after adding an appropriate amount of methyl alcohol solution into the raw material powder as a lubricant, the raw material powder was filled into a die and pressure of 686 Mpa was applied to form a compact. Next, in a heating furnace with a controlled mixed gas atmosphere of hydrogen and nitrogen, the compact was heated to be sintered for 60 minutes at 125 °C and then furnace-cooled. Next, after a 30-minute solution treatment under an argon gas atmosphere at 900 °C, the compact was thrown into oil for hardening, and in the same atmosphere at 200 °C, tempering was performed for 60 minutes. As a result, the steel in Embodiment 1 was obtained.

[Embodiment 2] Fe coarse powder + Fe fine powder, Ni 6 %

With the exception that the Ni content was changed to 6 wt%, the steel in Embodiment 2 was obtained in the same manner as in Embodiment 1.

[0020] [Comparative Example 1] Fe coarse powder, Ni 2 %

With the exception that atomized Fe powder (grain size: 74 μm or smaller) classified by -200 mesh was solely used, the steel in Comparative Example 1 was obtained in the same manner as in Embodiment 1.

[Comparative Example 2] Forging without heat treatment

With the exception of a forging treatment being conducted after sintering, the steel in Comparative Example 2 was obtained by performing the same heat treatment as in Embodiment 1. The specifications of Fe powder and Nickel powder in each embodiment and each comparative example above are shown in Table 1, and the composition of each element in Table 2.

[0021] [Table 1]

	Component (wt%)				
	Ni	Mo	Mn	C	Fe
Embodiment 1, Comparative Example 1, 2	2.0	0.5	0.2	0.4	Bal
Embodiment 2	6.0	0.5	0.2	0.4	Bal

[0022] [Table 2]

	Fe powder		Ni
	Coarse powder	Fine powder	Fine powder
Embodiment 1, 2	Atomized Fe powder	Carbonyl Fe powder	Carbonyl Ni powder

Comparative example 2	Grain size 53-74 $\mu\text{m}$	Average grain size 5 $\mu\text{m}$	Average grain size 5 $\mu\text{m}$
Comparative example 1	Atomized Fe powder Average grain size 74 $\mu\text{m}$		Carbonyl Ni powder Average grain size 5 $\mu\text{m}$

[0023] A. Mechanical properties, hardness, etc.

Regarding the embodiments and each comparative example above, a tensile test and a fatigue test were conducted to examine the mechanical properties. Furthermore, the hardness gradient was calculated by examining the relative density and the hardness of the Ni enriched phase and the matrix structure. The results are shown in Table 3.

[0024] [Table 3]

	Tensile strength (Mpa)	Fatigue strength (Mpa)	Durability limit rate (R)	Elongation (%)	Relative density (%)	Hardness (Hv)		Hardness gradient ( $\Delta\text{Hv}$ )
						R	M	
Emb.1	1400	410	0.29	2	94	665	615	50
Emb.2	1770	438	0.25	9	96	672	638	34
Comp. E.1	1300	360	0.28	2	91	662	575	87
Comp. E. 2	816	446	0.55	9	99	241		0

\* "R" in hardness is the hardness of the Ni enriched phase, "M" is the hardness of the matrix structure

[0025] According to Table 3, the hardness gradient of the steel in each embodiment based on the present invention is larger compared to the comparative examples, particularly proving the excellent tensile strength with respect to the mechanical properties. It has been ascertained that due to the large hardness gradient, inhomogeneous structures were generated and actions inhibiting crack propagation were fully promoted, thus enhancing the mechanical properties. Furthermore, in Comparative Example 2, since forging was performed after sintering, the structures were kneaded and, as a result, a Ni enriched phase was not generated. Therefore, the hardness and tensile strength became considerably lower compared to the rest.

[0026] B. An observation of the metallic structure

Next, with regard to the steel in the above Embodiment 1 and Comparative Example 1,

the internal structures were examined with a microscope. Figure 1 and Figure 2 respectively are photomicrographs showing the internal structure of each steel in Embodiment 1 and Comparative Example 1. In both, the matrix structure indicated by B is observed as a tempered martensite structure leaving the Ni enriched phase, indicated by the white area A, therein. However, it is understood that the Ni enriched phases in Embodiment 1 are smaller than those in Comparative Example 1 and are diffused in large numbers, making it more likely to inhibit crack propagation and resulting in enhancement of the steel strength.

[0027] Figure 3 and Figure 4 respectively are photomicrographs showing the fatigue crack propagation routes of each steel in Embodiment 1 and Comparative Example 1, with the black areas on the left side in the photographs being cracks. From these figures, the difference in fatigue crack propagation depending on the inhomogeneous structure is clear. That is, in Figure 3, the fatigue crack routes are propagating as if diverting the Ni enriched phases, indicating that a large amount of energy has been used for crack propagation. On the other hand, in Figure 4, although Ni enriched phases are observed, they are small in terms of quantity and size, and thus have little effect in preventing the cracks from propagating. Accordingly, in Figure 4, the cracks are likely to propagate easily through matrix structures which are easily broken. From this, it is clear that the structure shown in Figure 3 has higher strength compared to Figure 4.

[0028]

[Effect of the Invention] As explained, in the present invention, for producing high strength sintered alloy steel, composing Fe powder that is to be the main raw material powder with a combination of coarse powder and fine powder, and further regulating the composition of Ni along with other elements and the size of the powder accordingly, result in an effect of suitably manufacturing high strength sintered alloy steel that is excellent in mechanical properties such as tensile strength, fatigue strength, etc.

[Brief Description of the Drawings]

[Figure 1] A photomicrograph showing the internal structure of an embodiment of the present invention

[Figure 2] A photomicrograph showing the internal structure of a comparative example

[Figure 3] A photomicrograph showing fatigue crack propagation routes of an embodiment of the present invention

[Figure 4] A photomicrograph showing fatigue crack propagation routes of a comparative example

[Figure 1] A photograph substituting a drawing

[Figure 2] A photograph substituting a drawing

[Figure 3] A photograph substituting a drawing

[Figure 3] A photograph substituting a drawing

Continued from the front page

F terms (reference)

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